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INVESTIGATION OF  
MECHANISMS FOR  
ELECTROCHEMICAL CATALYSIS

SECOND QUARTERLY REPORT

*By*

*J. R. Aylward—S. W. Smith*

FEBRUARY 1967

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February 1967 .

INVESTIGATION OF MECHANISMS FOR  
ELECTROCHEMICAL CATALYSIS

Quarterly Report

August 1, 1966 to October 31, 1966

Report No. 2

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**FOREWORD**

This report was produced in accordance with U. S. Army Contract No. DA 28-043-AMC-02205(E) by the Pratt & Whitney Aircraft Division of the United Aircraft Corporation. It discusses the work performed during the period from 1 August 1966 through 31 October 1966.

## ABSTRACT

A program is being conducted to investigate the mechanisms of electrocatalysis with respect to the anodic oxidation of selected fuels by novel electrocatalysts in acid electrolyte at temperatures below 150°C and at ambient pressure. Most of the effort is on select transition metal silicides surface-doped with trace amounts of platinum and/or gold.

Some additional materials (transition metal borides) were screened for use as possible electrocatalysts. The electrochemical behavior of TaSi<sub>2</sub> in acid electrolyte was studied by measuring the polarization via triangular potential sweep and steady-state methods. The catalytic activity of TiSi<sub>2</sub> and MoSi<sub>2</sub> surface-doped with platinum and gold by various techniques was investigated.

None of the new materials screened showed sufficient corrosion resistance in acid for use as catalysts or supports. TaSi<sub>2</sub> in the absence of fuel gave a relatively high cathodic current in the potential range from zero to 200 mV which is difficult to explain on the basis of known reactions. MoSi<sub>2</sub> was not amenable to surface-doping but this technique applied to TiSi<sub>2</sub> gave promising catalytic activity for the oxidation of hydrogen. Platinum and gold deposited simultaneously in trace amounts on TiSi<sub>2</sub>, resulted in higher catalytic activity for hydrogen oxidation than the deposition of platinum alone or gold deposition followed by platinum deposition.

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## I. INTRODUCTION

A program is being conducted to investigate the mechanisms of electrocatalysis with respect to the anodic oxidation of selected fuels by novel electro-catalysts in acid electrolyte at temperatures below 150°C and at ambient pressure. These studies are especially concerned with how the surface properties influence the catalytic activity and reaction mechanism. The study is being conducted in order to investigate active, low-cost catalysts for direct energy conversion devices such as fuel cells.

The program is composed of the selection and screening of non-noble materials for corrosion resistance and electrochemical activity followed by a detailed investigation of selected transition metal silicides for the electrochemical oxidation of fuels. The effect of adding trace amounts of materials such as gold and platinum to the surface of the silicides is also being investigated.

The results obtained during the first quarter showed no evidence that hydrogen can be absorbed or oxidized on a pure transition-metal silicide surface. Combining silicides with trace amounts of gold and/or platinum resulted in interesting catalytic activity, and activating the surface of  $TiSi_2$  with trace amounts of platinum gave considerable activity for hydrogen oxidation.

The surface oxides of  $TiSi_2$ ,  $MoSi_2$  and  $VSi_2$  were found to be good electronic conductors and their formation was reversible up to 1.2 V. Below 400 mV the steady state corrosion rate of  $MoSi_2$  and  $TiSi_2$  at 50°C is approximately  $10^{-5}$  amp.  $cm^{-2}$ , based on geometrical area. The corrosion current of  $VSi_2$ , however, increases rapidly at potentials above 200 mV.

## II. PROGRESS

### A. SCREENING OF MATERIALS FOR ELECTROCATALYSIS

The following materials in the form of -325 mesh powders were purchased from City Chemical Corporation, New York City, for screening as possible electrocatalysts or supports: MoN, BP, CrC, MoC, and the diborides of Cr, Zr, Ti, Mo, and Ta.

A different cell and electrode design than that previously described for material screening was used as shown in Figure 1. This is a three-compartment cell

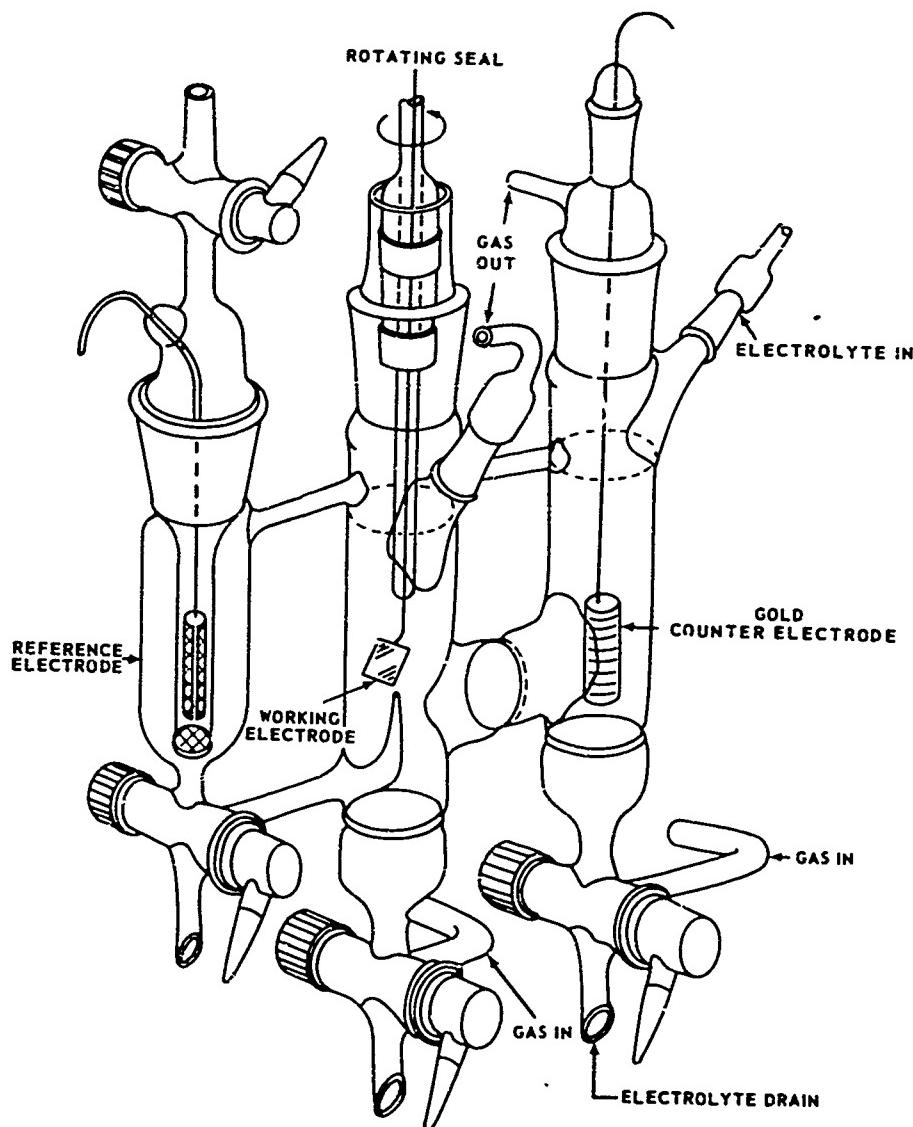


Figure 1      Three-Compartment Cell Used for Screening Studies

with a fritted glass disk between working electrode and counter-electrode compartments. The reference electrode compartment terminates in a Luggin capillary beneath the working electrode. The working electrode passes into the cell through a rotating, water-sealed joint and is rotated at 600 rpm by a synchronous motor. The counter electrode is a 2 x 2 cm gold sheet. Gases are admitted into the cell electrolyte through glass frits in the bottoms of the working and counter electrode compartments.

The electrode was a one  $\text{cm}^2$ , rotating (600 rpm) flag-type, inclined approximately 45° from the axis of rotation (Figure 1). These electrodes were prepared by spraying a water slurry of the powdered (-325 mesh) material to be tested and Teflon 30 (20 wt. %) onto a 100 mesh gold screen current collector, wet pressing at 50 psi, followed by a sinter at 250°C for 5 minutes. The loading was approximately 50 mg/cm<sup>2</sup>.

The experimental conditions and auxiliary equipment were as described in the First Quarterly Report. Corrosion was measured in a nitrogen-saturated 1 M  $\text{H}_2\text{SO}_4$  at 50°C from zero to 1.0 volt\* at 50 mV intervals with current readings taken after the desired potential had been maintained for 10 minutes.

The BP and CrC powders were not tested for corrosion behavior because both showed a high electronic resistance. TaB<sub>2</sub> exhibited good corrosion characteristics ( $< 10^{-5}$  amp/cm<sup>2</sup> up to 0.8 volts) but no detectable activity for hydrogen or methanol oxidation in 1 M  $\text{H}_2\text{SO}_4$  at 50°C. All of the other materials tested (MoN, MoC, and the diborides of Cr, Zr, Ti, Ni and Mo) gave corrosion currents  $> 10^{-4}$  amp/cm<sup>2</sup> and were therefore not evaluated for catalytic activity.

## B. KINETIC STUDIES OF TRANSITION METAL SILICIDES

The preliminary kinetic studies on  $\text{TiSi}_2$  and  $\text{MoSi}_2$  were continued during this period with emphasis on studying the oxidation of hydrogen at platinum and platinum-gold doped surfaces. Untreated  $\text{TaSi}_2$  has also been examined. The experimental cell and equipment were described in detail in the First Quarterly Report. The electrode is a cylindrical rod inserted in Teflon tubing so that only the end (geometrical area 0.5 cm<sup>2</sup>) is available to the electrolyte. The Teflon tube also served to connect the rod to a glass tube from the 600 rpm stirring motor which rotated the electrode. The electrode was treated with 2.7 molar HF-14 molar  $\text{HNO}_3$  solution at 90°C for 1 minute followed by washing in triple-distilled water. This treatment results in a surface which is macroscopically smooth but microscopically rough and porous. Since it has not been possible to measure the true surface area of the sample, measured currents are reported rather than attempting to estimate current densities, and additions of platinum or gold are given in total weight added, rather than on a surface-coverage basis.

\*all potentials refer to an ambient temperature hydrogen electrode in the same solution.

Before measurements were made on the electrode, it was held at -600 mV for 30 minutes, and returned to this potential between potential sweeps in order that the oxide layer reach a small constant thickness for all experiments. The triangular potential sweeps at 34 mV/sec were made between -600 and 1100 mV. Triangular potential sweeps at 100 mV/min were generally made between -50 and 500 mV after stepping the potential from -600 mV. A few of the slow sweeps were made from -600 to 1100 mV and showed no major departure from the sweeps started at -50 mV.

### 1. Preliminary Data on TaSi<sub>2</sub>

The electrochemical behavior of TaSi<sub>2</sub> has been investigated under the same conditions as reported in the First Quarterly Report for the disilicides of titanium, molybdenum and vanadium. The potential sweep and steady state (15-minute points) data for TaSi<sub>2</sub> shown in Figures 2, 3 and 4, indicate no

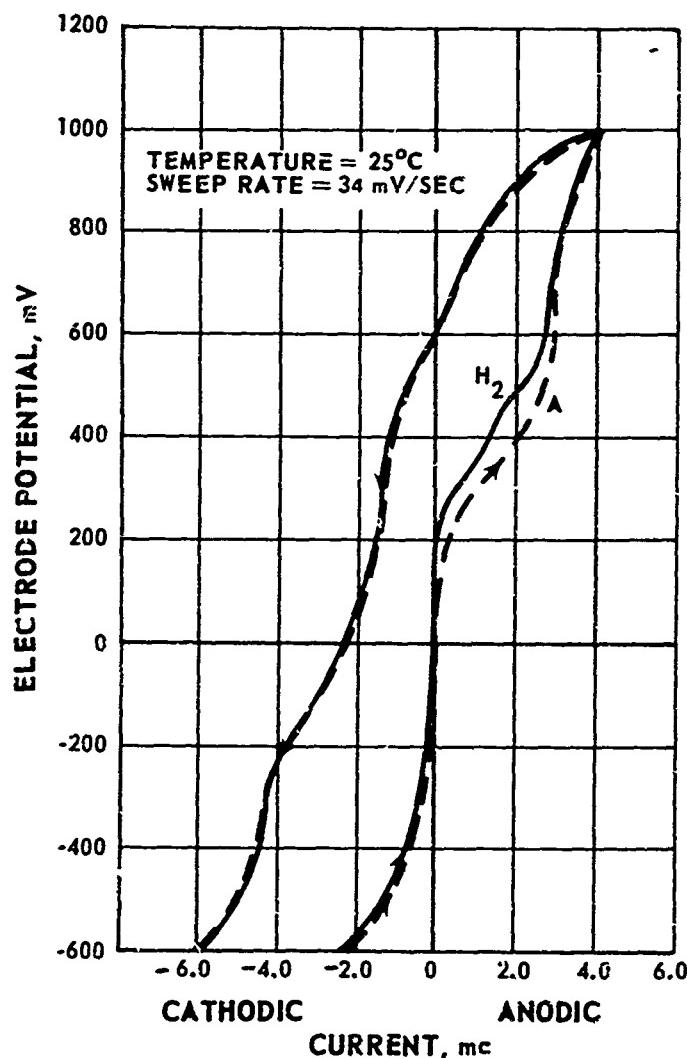


Figure 2      Triangular Potential Sweep Curves for TaSi<sub>2</sub> with Sweep Rate of 34 mV/sec.

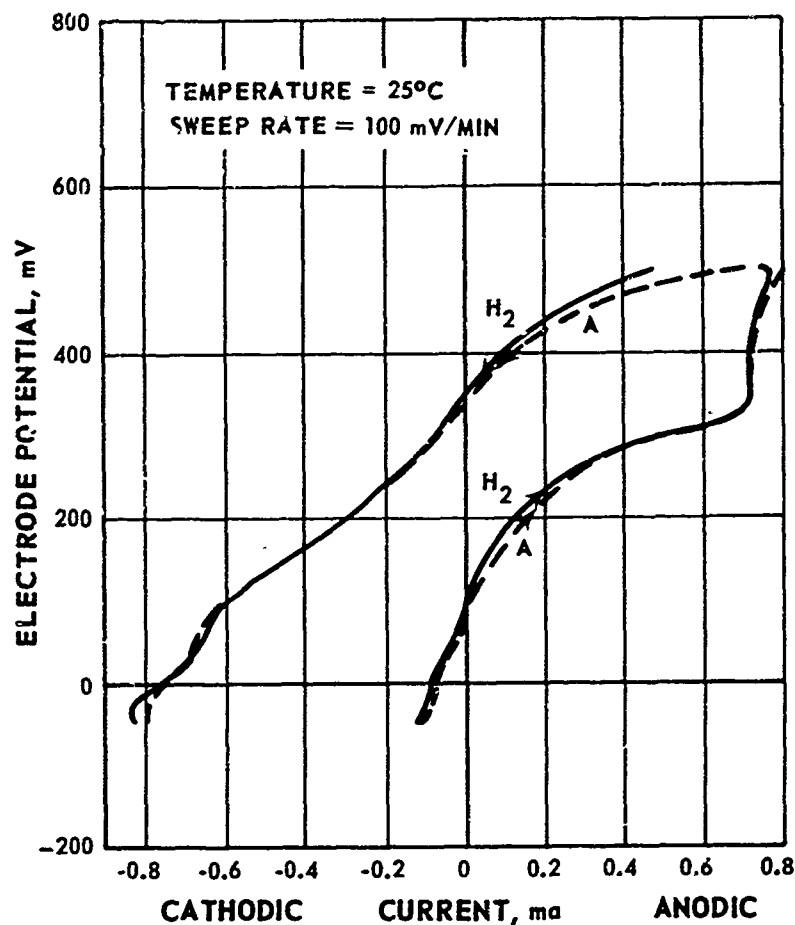


Figure 3      Triangular Potential Sweep Curves for  $\text{TaSi}_2$  with Sweep Rate of 100 mV/min.

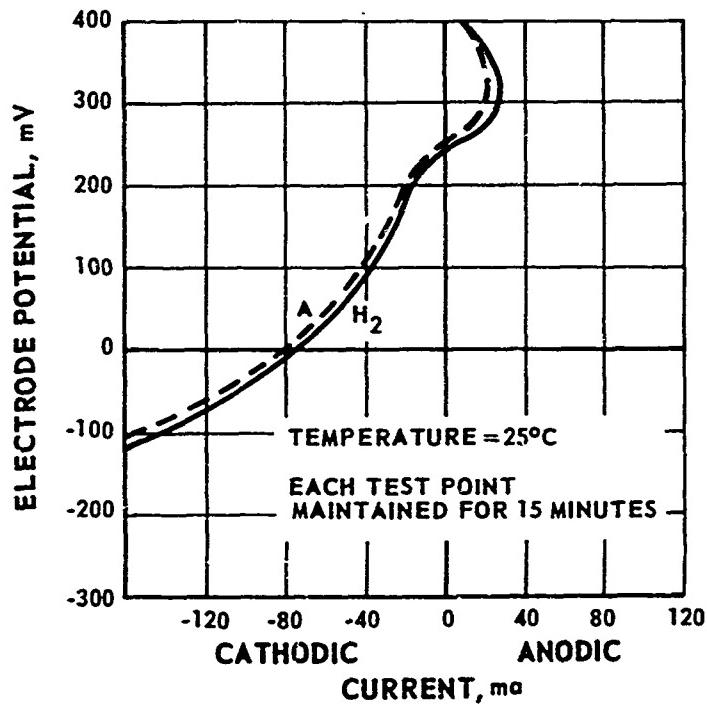


Figure 4      Steady-State I-E Data for  $\text{TaSi}_2$ .

essential difference between solutions saturated with argon and hydrogen. The steady-state data also shows relatively large cathodic current in the range of interest for hydrogen oxidation, which is difficult to explain with present data. Further investigations will be carried out in an attempt to determine the cause of this cathodic current and to determine if platinum or gold-platinum surface-doping has any effect.

## 2. Surface Activation of $TiSi_2$ with Platinum and Gold-Platinum

A series of platinum additions, from  $0.06 \mu\text{g}$  to  $6.4 \mu\text{g}$ , was made to a  $TiSi_2$  electrode. The electrode was held at  $-600 \text{ mV}$  for 30 minutes after the required amount of platinum solution had been added, and then triangular potential sweeps between  $-600 \text{ mV}$  and  $1100 \text{ mV}$  were made at  $34 \text{ mV/sec}$  and  $100 \text{ mV/min}$ . From the sweep curves it was obvious that an impurity was present in the solution which absorbed at low potentials and poisoned the electrode for hydrogen oxidation. The impurity was removed above  $300 \text{ mV}$  and was re-absorbed only slightly on the back sweep. The results of the  $1100$  to  $-600 \text{ mV}$  portion (back sweep) of the  $34 \text{ mV/sec}$  sweep, shown in Figure 5, indicate that the activity increases rapidly with increasing platinum loading at low loading levels and approaches a limit at about  $4 \mu\text{g}$  for a geometrical area of  $0.5 \text{ cm}^2$ .

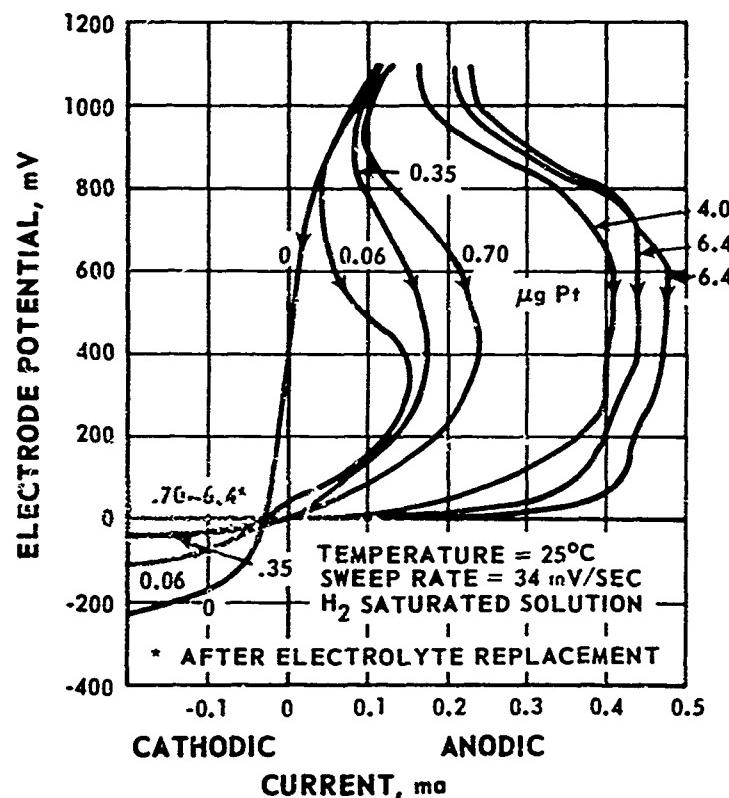


Figure 5 Potential Sweep Curves for Platinum Activated  $TiSi_2$ .

After the above series was completed, the electrode was held at 800 mV while the solution was withdrawn from the cell and fresh electrolyte added. A comparison of the results obtained using the original electrolyte and the fresh electrolyte is shown in Figure 6, where the complete triangular

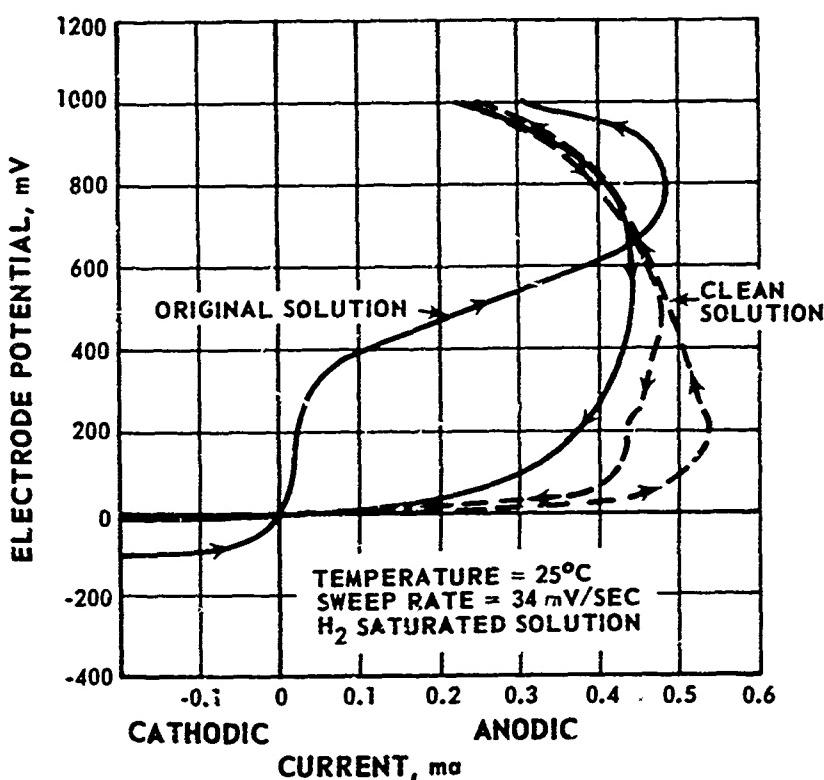


Figure 6  
Effect of Solution Replacement on Triangular Potential Sweep  
Curves for Platinum Activated  $\text{TiSi}_2$ .

sweep at 34 mV/sec is shown for both cases. It is apparent that some residual effect of the impurity remained on the back sweep in the original electrolyte and that the impurity could be removed with the solution, since no evidence of it is seen in the second sweep curve. If the electrode is now allowed to remain at -600 mV for 2 hours, some evidence of poisoning reappears, indicating that a small amount of impurity remained in the system. Although this problem did not occur in subsequent experiments, it has been considered necessary to modify the experimental set-up somewhat to prevent repetition of this problem in the future.

A sample of  $\text{TiSi}_2$  was plated with 1.5  $\mu\text{g}$  of gold from 4.5 M  $\text{H}_2\text{SO}_4$  containing the appropriate amount of gold sulfate, followed by activation with platinum in several increments. The results of this experiment are shown in Figure 7. These data show a small amount of activity for the  $\text{TiSi}_2$ -Au combination in the absence of platinum. A comparison of Figure 7 with

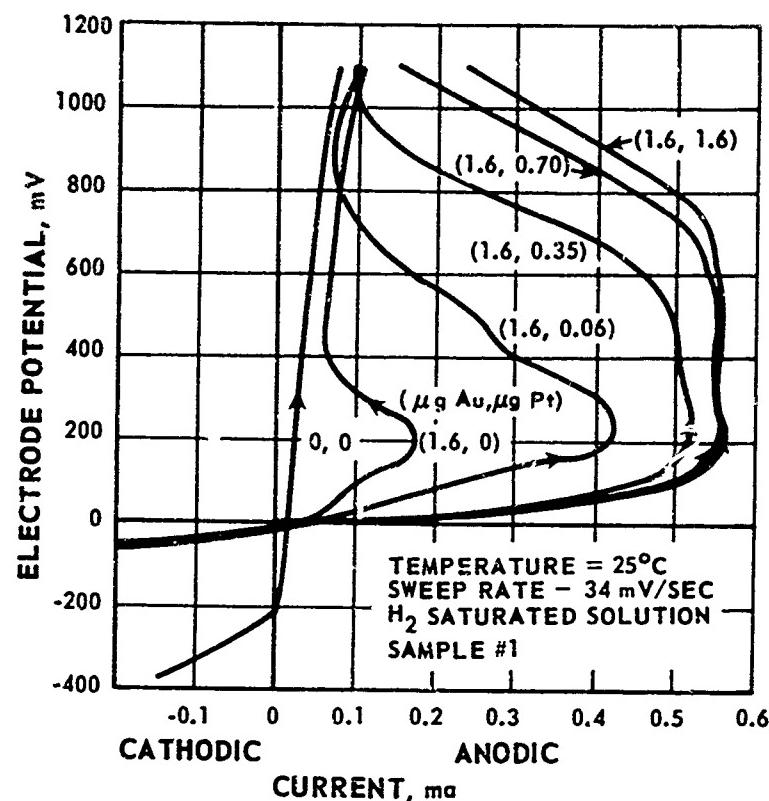


Figure 7 Potential Sweep Curves for Gold-Platinum Activated TiSi<sub>2</sub>.

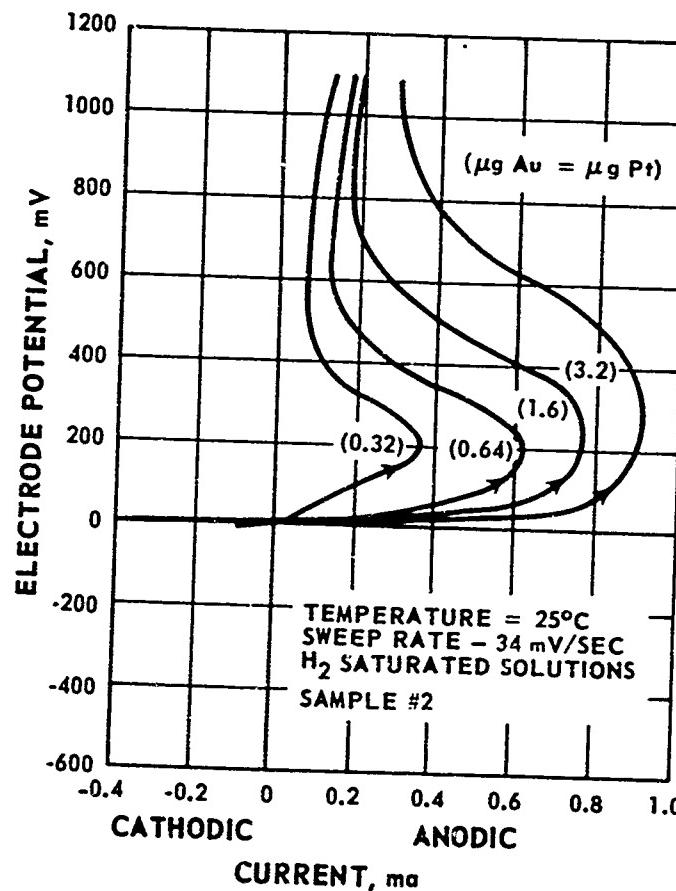


Figure 8 Potential Sweep Curves for TiSi<sub>2</sub> Activated by Co-Deposition of Gold and Platinum.

Figure 5 shows that the presence of the gold considerably enhances the activity over that for doping with platinum alone.

Another sample of  $\text{TiSi}_2$  was activated by plating with gold and platinum simultaneously from a common sulfate bath which contained essentially equal amounts of each metal. Since the rate at which each would plate was not known, the required total amount of the metals for each increment was added to the bath and the plating was carried to completion. The results of this experiment are shown in Figure 8. The co-deposit shows a higher current at equivalent platinum loadings than either of the previous experiments (compare with Figures 5 and 7). It was noticed, in addition, that the appearance of the plate is much different in the case of simultaneous plating. The previous deposits did not appreciably affect the appearance of the silicide surface, while the co-deposit was clearly seen as a poorly adherent black layer.

A summary of the data on platinum and platinum-gold is shown in Figure 9, where the current at 50 mV in  $\text{H}_2$  saturated solution is plotted against the platinum loading on the electrode. This plot shows clearly that the

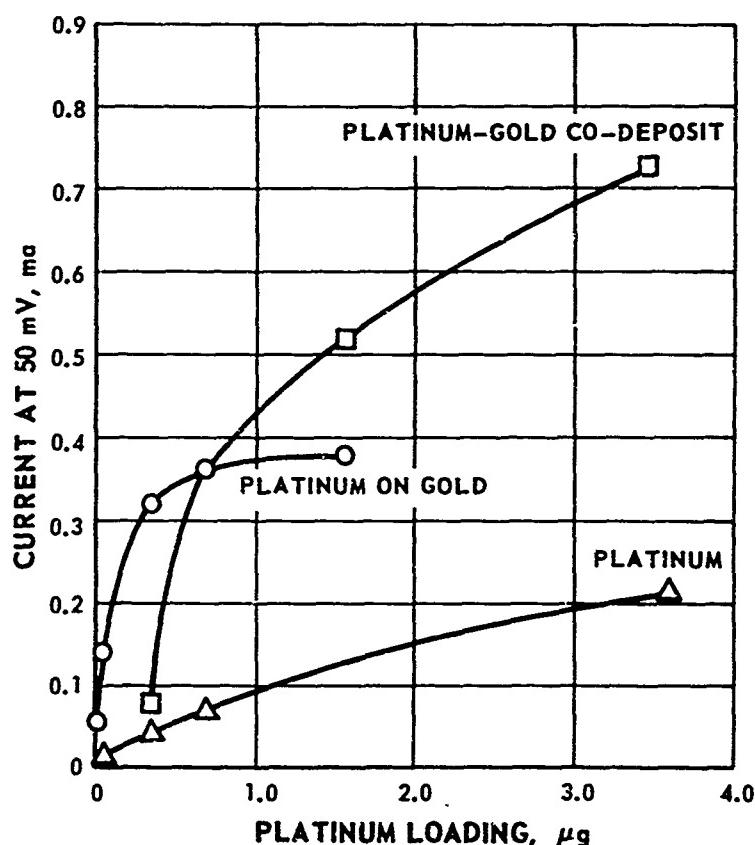


Figure 9 Performance at 50 mV of Activated  $\text{TiSi}_2$  Electrodes as a Function of Pt Loading.

pre-plating of the silicide surface with gold enhances the activity at equivalent platinum loadings, and that the co-deposition of gold and platinum results in the highest activity.

3. Surface Activation of MoSi<sub>2</sub>, with Platinum and Gold-Platinum

Samples of MoSi<sub>2</sub> were treated with platinum and platinum-over-gold in the same manner as the TiSi<sub>2</sub> samples. The results show that although the platinum is deposited, as evidenced by overvoltage changes in the hydrogen evolution region, no activity for the oxidation of hydrogen could be observed except at high ( $> 5 \mu\text{g}$ ) platinum loadings, in which case it was marginal. In the case where platinum was deposited after gold pre-treatment, some activity was found; however, it was less than one-half that obtained for TiSi<sub>2</sub>. It is difficult to understand why the oxidation rate of hydrogen is negligible on an electrode which catalyzes the hydrogen evolution reaction at an appreciable rate. This phenomenon will be investigated further in the hope of revealing some aspects of the mechanism of catalysis with these materials.

4. X-ray Analysis of Ti, Mo, and Ta Silicides

Samples (0.328 cm thick) were cut from the end of each of the silicide rods after the potential sweep and steady state data were completed. The samples were subjected to X-ray analysis on both sides. The results from the side which had been used as the electrode surface are presented in Table I, and show that the samples have the intended composition, except for a small Si content in the TiSi<sub>2</sub>. The side corresponding to the saw cut did not yield a readable pattern. The thickness of the oxide film on the surface is too thin to be detected by this method.

5. Investigations of the Surface Oxides on MoSi<sub>2</sub> and TiSi<sub>2</sub>

An experiment was conducted to determine the potential at which the surfaces of MoSi<sub>2</sub> and TiSi<sub>2</sub> are free of oxide layers in 1 M H<sub>2</sub>SO<sub>4</sub>. The samples were held in an inverted position in 1 M H<sub>2</sub>SO<sub>4</sub> with a small drop of mercury placed on the electrode surface. It was assumed that when the surface was free of oxide the mercury would wet the electrode. To effect surface-oxide reduction, the potential was lowered in 100 mV increments to -500 mV with each potential held for 10 minutes while the mercury drop was observed, but no change in contact angle could be detected for this potential region. The potential was then lowered to -600 mV for 30 minutes followed by -1400 mV for 30 minutes. Even at -1400 mV no significant change in contact angle between the mercury drop and electrode surface

could be observed for either  $\text{MoSi}_2$  or  $\text{TiSi}_2$ . Thus, it appears that the oxide on these two silicides cannot be completely removed by electrochemical reduction in 1 M  $\text{H}_2\text{SO}_4$  at potentials down to -1400 mV. Obviously when these silicides are surface-doped with noble metals at -600 mV, the metals are deposited on an oxide surface rather than the bare silicide.

A workable means of measuring the amount of oxide formed on the surface has not been found. The problem is complicated by the current due to hydrogen evolution below 0 mV, and the variation of this current as the oxide thickness changes with potential and time.

TABLE I  
DATA FROM X-RAY ANALYSIS OF SILICIDE ROD SAMPLES

<u>Compound</u>	<u>Phases Present</u>	<u>Measured Density</u>	<u>X-ray Density</u>	<u>% Theoretical Density</u>
$\text{MoSi}_2$	major phase $\text{MoSi}_2$ all lines identified	6.002	6.276	95.7
$\text{TaSi}_2$	major phase $\text{TaSi}_2$ all lines identified	8.226	9.077	90.6
$\text{TiSi}_2$	major phase $\text{TiSi}_2$ second weak phase Si two additional lines identified	3.850	3.849	~100

### III. CONCLUSIONS AND RECOMMENDATIONS

Of the silicides investigated to date,  $TiSi_2$  appears to be the most likely candidate for the detailed kinetic investigation, by reason of its low corrosivity in the potential range from 0 to 800 mV and the ease with which it may be activated with Pt or Pt-Au. Therefore, unless  $WSi_2$  shows unexpectedly superior characteristics when it is received from the vendor, further work will be done on  $TiSi_2$ .

It is also clear that the activity for hydrogen oxidation on  $TiSi_2$  is considerably enhanced in the lower-platinum-coverage range by a pre-activation with gold and that essentially no activation of  $MoSi_2$  is possible without a gold activation step. It further appears that a co-deposit of platinum and gold has distinctly different characteristics than that applied by individual plating steps. No attempt has been made as yet to vary either the composition or character of the co-deposit. The combination of platinum and gold activations, while it has not yet been shown to give results superior to platinum itself, at least results in a very efficient use of the platinum.

The results obtained show that additional precautions must be taken to further reduce the possibilities for contamination of the electrolyte. This will be particularly important in the investigation of methanol and hydrocarbon oxidation where impurities may interfere to a larger extent. Steps are now being taken to modify the cell, purification train, and electrolyte supply, in order to reduce the possibility of contamination. In addition to this, the entire system is being installed in a clean box.

The silicide samples which are being used have been shown by X-ray analysis to be essentially of the intended composition (except for a small amount of Si in the  $TiSi_2$  sample).

During the third quarter the sample of  $WSi_2$  now on order from the vendor will be tested under the same conditions as have the previous silicides, and additional investigations will be done on  $TaSi_2$  in an attempt to elucidate the cause of the cathodic current at potentials above zero volts. Most of the effort, however, will be concentrated on investigating the oxidation of fuels on  $TiSi_2$  activated with trace amounts of noble metals with the purpose of developing the activation technique for optimum noble metal utilization. Experiments are also planned which will lead to a more quantitative expression of the noble metal utilization.

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13. ABSTRACT <p>A program is being conducted to investigate the mechanisms of electrocatalysis with respect to the anodic oxidation of selected fuels by novel electrocatalysts in acid electrolyte at temperatures below 150°C and at ambient pressure. Most of the effort is on select transition metal silicides surface-doped with trace amounts of platinum and/or gold.</p> <p>Some additional materials (transition metal borides) were screened for use as possible electrocatalysts. The electrochemical behavior of TaSi<sub>2</sub> in acid electrolyte was studied by measuring the polarization via triangular potential sweep and steady-state methods. The catalytic activity of TiSi<sub>2</sub> and MoSi<sub>2</sub> surface-doped with platinum and gold by various techniques was investigated.</p> <p>None of the new materials screened showed sufficient corrosion resistance in acid for use as catalysts or supports. TaSi<sub>2</sub> in the absence of fuel gave a relatively high cathodic current in the potential range from zero to 200 mV which is difficult to explain on the basis of known reactions. MoSi<sub>2</sub> was not amenable to surface-doping but this technique applied to TiSi<sub>2</sub> gave promising catalytic activity for the oxidation of hydrogen. Platinum and gold deposited simultaneously in trace amounts on TiSi<sub>2</sub>, resulted in higher catalytic activity for hydrogen oxidation than the deposition of platinum alone or gold deposition followed by platinum deposition.</p>		

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